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IS 258 (2000): Potash Alum [CHD 1: Inorganic Chemicals]

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भारतीय मानक

पोटाश एलम — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

POTASH ALUM — SPECIFICATION

(Second Revision)

ICS 71.060.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 2000

Price Group 5

General Inorganic Chemicals Sectional Committee, CHD 03

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

The potash alum is used in dyeing, printing, fabrics, paper, vegetable glue, purification of water, as cattle lick and in the manufacture of dyes. Alum is also used as a local astringent, gargle or spray solution, styptic to bleeding points and for various other pharmaceutical applications. It is also widely used in analytical and research laboratories.

Initially two Indian Standard specifications for potash alum had been published, IS 258 : 1950 for technical grade and IS 378 : 1952 for pharmaceutical grade of the material. In the first revision of the standard, the two Indian Standards were amalgamated while including another grade, namely, analytical reagent. Consequently, IS 378 : 1952 covering the pharmaceutical grade was withdrawn. Changes were also made in the sampling procedure and some of the methods of test. Additional requirements for photographic industry were also incorporated.

In the present (second) revision, alum of Pharmaceutical grade has been deleted as the same is covered in Indian pharmacoepia 1996. Further EDTA method for determination of potash alum has been modified. The requirements of copper and zinc has been deleted.

There is no ISO specification on this subject.This standard has been prepared based on indigenous manufacturers' data prevalent in the field in india.

Composition of the Committee responsible for formulating this standard is given in Annex K.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
POTASH ALUM — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for potash alum (aluminium potassium sulphate).

2 NORMATIVE REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
321 : 1964	Absolute alcohol (*revised*)
460 : 1985	Test sieves : Part 1 Wire cloth test sieves (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
4905 : 1968	Methods for random sampling

3 GRADES

3.1 The material shall be of the following two grades:

a) *Technical* (TECH) — used in dyeing, printing fabrics, paper, vegetable glue, purification of water, as cattle lick and in the manufacture of dyes and lakes.

b) *Analytical Reagent* (AR) — used in analytical and research laboratories.

3.1.1 Special requirements for photographic industry have been included in technical grade (*see* Table 1).

4 REQUIREMENTS

4.1 Description

The material shall be in the form of colourless, transparent crystals or crystalline lumps or a white powder free from all visible impurities. The material shall be soluble in glycerine. It shall not dissolve in rectified spirit.

4.2 Solubility in Water

For analytical reagent grades, 50 g of the material, when dissolved in 500 ml of water, shall give a clear, colourless solution.

4.3 The material shall also comply with the requirement given in Table 1 when tested according to the method prescribed in Annexes A to H. References to the relevant Annexes is given in col 5 of Table 1.

5 PACKING AND MARKING

5.1 Packing

For bulk packing, the material shall be packed in flexible containers, such as of paper, plastic, fibreboard and jute with suitable liner. For smaller packing, the material shall be packed in moisture-proof glass polythene bottles.

5.1.1 For special requirements, the packing may be as agreed to between the purchaser and the supplier.

5.2 Marking

Each packages shall be marked with the following information:

a) Name and grade of the material,

b) Mass of the material,

c) Indication of the source of manufacture,

d) Month and year of manufacture, and

e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

5.2.1 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the

Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative samples of the material, the number of tests and the criteria for conformity shall be as prescribed in Annex J.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Potash Alum

(*Clauses* 3.1.1 *and* 4.3)

Sl No.	Characteristic	Requirements for Grade		Method of Test, Ref to Annex
		Tech	AR	
(1)	(2)	(3)	(4)	(5)
i)	Potash alum [$Al_2(SO_4)_3K_2SO_4$ $24H_2O$], percent by mass, *Min*	99.0	99.50	A
ii)	Matter insoluble in water percent by mass, *Max*	0.25	—	B
iii)	Soluble iron compounds (as Fe) percent by mass, *Max*	0.007	0.001	C
iv)	Ammonium salts (as NH_3) percent by mass, *Max*	0.20	0 .025	D
v)	*p*H of 5 percent solution, not less than	3	3	E
vi)	Arsenic (as As_2O_3), parts per million, *Max*	8	—	F
vii)	Heavy metals (as Pb), parts per million, *Max*	50	10	G
viii)	Chlorides (as Cl), percent by mass, *Max*	—	0.02	H

NOTE — For photographic industry, the requirements of potash alum shall be 99.5 percent by mass, *Min* and matter insoluble in water shall be 0.05 percent by mass, *Max*.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF POTASH ALUM [$Al_2(SO_4)_3K_2SO_4\ 24H_2O$]

A-1 PREPARATION OF THE TEST SAMPLE

A-1.1 Procedure

To obtain the *prepared sample* crush about 200 g of the material to pass 1.00 mm IS Sieve (*see* IS 460). Keep this *prepared sample* in a clean glass-stoppered weighing bottle in a dessicator for test purposes.

A-2 GENERAL

Two methods have been prescribed for determination of potash alum gravimetric method (Method A) and titration with EDTA method (Method B).

A-2.1 Method A (Gravimetric Method)

A-2.1.1 *Outline of the Method*

Potash alum is estimated by precipitating aluminium with ammonium hydroxide and calculating the mass of potash alum from the ignited residue of aluminium oxide.

A-2.1.2 *Reagents*

A-2.1.2.1 *Ammonium chloride*

Solid.

A-2.1.2.2 *Dilute sulphuric acid*

Approximately 1 N.

A-2.1.2.3 *Methyl red indicator*

Dissolve 0.30 g of water soluble methyl red in water and dilute it to one litre.

A-2.1.2.4 *Dilute ammonium hydroxide*

Approximately 5 N.

A-2.1.2.5 *Ammonium nitrate solution*

Approximately 2 percent (*m/v*).

A-2.1.3 *Procedure*

Weigh accurately about 2 g of the *prepared sample* and dissolve in 300 ml of water. Filter to remove any undissolved residue and thoroughly wash the filter paper with hot water. Add to the filtrate and washings about 5 g of ammonium chloride and 10 ml of dilute sulphuric acid. Boil the solution and add two drops of methyl red indicator. Add ammonium hydroxide solution, drop by drop, till the colour changes to distinct yellow. Boil for two minutes and filter immediately through Whatman filter paper No. 41 or its equivalent. Wash the precipitate of mixed oxides of iron and aluminium with hot ammonium nitrate solution till the filtrate is free from chlorides. Ignite the precipitate to a temperature of 1 100 to 1 200°C in a tared silica crucible to constant mass.

A-2.1.4 *Calculation*

Potash alum, percent by mass = $9.307\,(A-1.429\,7B)$

where

A = percent by mass of the ignited residue; and

B = percent by mass of iron as determined under Annex C.

A-2.2 Method B (EDTA Method)

A-2.2.1 *Reagents*

A-2.2.1.1 *Disodium ethylene diamine tetra acetate (EDTA) Solution*

0.05M. Dissolve 18.612 5 g of disodium ethylene diamine tetra acetate dihydrate in water and make the volume to one litre in a volumetric flask.

A-2.2.1.2 *Hexamine (Hexamethylene tetramine) analar*

A-2.2.1.3 *Xylenol orange indicator*

Prepare 0.5 percent in water and add 1 to 2 drops of dilute sodium hydoxide solution.

A-2.2.1.4 *Standard lead nitrate solution*

0.05 M.

A-2.2.1.5 *Concentrated sulphuric acid*

A-2.2.2 *Procedure*

Dissolve 5 g of *prepared sample* (*see* **A-1**) in water. Acidify it by adding 1 to 2 ml of concentrated sulphuric acid and make up the volume to 500 ml in a volumetric flask. Take an aliquot of 50 ml into a conical flask, add 50 ml of 0.05 M EDTA solution and heat the content to just boiling. Cool it and add hexamine to adjust the *p*H to 5. Add few drops of xylenol orange indicator and titrate the excess EDTA solution with standard lead nitrate solution until the colour changes from yellow to pink.

A-2.2.3 *Calculation*

Calculate the potash alum content of the material on the assumption that one millilitre of 0.05 M EDTA solution is equivalent to 0.023 72 g of potash alum as $[Al_2(SO_4)]_3\ K_2SO_4\,24H_2O$.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF MATTER INSOLUBLE IN WATER

B-1 PROCEDURE

Weigh accurately about 25 g of the prepared sample. Dissolve in 250 ml of water by warming, if necessary, and by stirring the solution well. Filter through a tared filter paper or Gooch crucible or a sintered glass crucible (G No. 4). Wash the residue thoroughly with water till it is free from all soluble compounds, and dry to constant mass at 110 ± 5 °C.

B-2 CALCULATION

Insoluble matter, percent by mass = $\frac{m}{M} \times 100$

where

m = mass in g of the residue obtained, and

M = mass in g of the prepared sample taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF SOLUBLE IRON COMPOUNDS (as Fe)

C-1 OUTLINE OF THE METHOD

Iron is determined colorimetrically by visual comparison of the colour in Nessler tubes, using potassium thiocyanate.

C-2 APPARATUS

C-2.1 Nessler Tubes

50 ml capacity.

C-3 REAGENTS

C-3.1 Concentrated Nitric Acid

See IS 264.

C-3.2 Ammonium Persulphate

Solid.

C-3.3 Butanolic Potassium Thiocyanate

Dissolve 10 g of the potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml, and shake vigorously until the solution is clear.

C-3.4 Dilute Sulphuric Acid

Approximately 10 percent (*v/v*).

C-3.5 Standard Iron Solution

Weigh 0.702 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4 6H_2O$] and dissolve in 10 ml of dilute sulphuric acid. Dilute with water to make up the volume to 1 000 ml. Transfer 10 ml of this solution and again dilute with water to make up the volume in 100 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

C-4 PROCEDURE

Weigh accurately about 1 g of the prepared sample, dissolve it in water, and make up the volume to 100 ml. Pipette out exactly 10 ml of this solution into a beaker, add 1 ml of nitric acid and boil. Cool, transfer the solution to a Nessler tube, add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 s and allow the liquid to separate. Carry out a control test in the other Nessler tube, adding slowly from a burette a quantity of the standard iron solution, say V ml, in place of the material, and the same quantities of other reagents in the same total volume of the reaction mixture until the colour of butanol layer in the two tubes are exactly matched.

C-5 CALCULATION

$$\text{Iron (as Fe), percent by mass} = \frac{0.01\,V}{M}$$

where

V = volume in ml of standard iron solution required in the control test; and

M = mass in g of the material taken for the test.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF AMMONIUM SALTS (as NH_3)

D-1 OUTLINE OF THE METHOD

Ammonium salts are determined colorimetrically by visual comparison of the colour, using Nessler solution.

D-2 APPARATUS

D-2.1 Nessler Tubes

50 ml capacity.

D-3 REAGENTS

D-3.1 Sodium Hydroxide Solution

Approximately 10 percent (*m/v*).

D-3.2 Potassium Iodide

Crystals.

D-3.3 Mercuric Chloride Solution

Saturated.

D-3.4 Potassium Hydroxide

Solid.

D-3.5 Nessler Solution

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly, with stirring, mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide, and when it has dissolved, add one millilitre more of the mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle over-night, decant the clear solution and keep the solution in bottles closed with well-fitting rubber stoppers.

D-3.6 Standard Ammonia Solution

Dissolve 3.141 g of ammonium chloride in ammonia-free water and make up the volume to 1 000 ml. Dilute 100 ml of this solution to 1 000 ml with ammonia free water. One millilitre of this solution contains 0.01mg of ammonia (as NH_3).

D-4 PROCEDURE

Weigh accurately about 1 g of the prepared sample into a volumetric flask of 1 000 ml capacity and dissolve in ammonia-free water. Make up the volume to 1 000 ml with ammonia free water. Pipette out exactly 10 ml of this solution into a Nessler tube and dilute with 20 ml of ammonia free water. Add 5 ml of sodium hydroxide solution and one millilitre of Nessler solution. Make up the volume to 50 ml with ammonia-free water. Carry out a control test in the other Nessler tube, using 2 ml of standard ammonia solution in place of the material in case of the technical grade, and 1 ml in case of analytical reagent grade, the same quantities of other reagents in the same total volume of the reaction mixture. Compare the intensity of colour produced in the two tubes after 5 min.

D-4.1 The limit prescribed for ammonium salts in Table 1 shall be taken as not having been exceeded if any colour, produced in the test with the material, is not deeper than that produced in the control test.

ANNEX E

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF *p*H

E-1 PROCEDURE

Weigh 2.5 g of the prepared sample and dissolve in 50 ml of water. Determine the *p*H of the solution with the help of a suitable *p*H meter, using glass electrodes.

ANNEX F

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF ARSENIC

F-1 PROCEDURE

Dissolve 1.0 g of the prepared sample in 20 ml of water. Carry out the test for arsenic as prescribed in IS 2088, using for comparison a stain obtained with 0.008 mg of arsenic trioxide as (As_2O_3) in case of technical grade of the material.

F-2 The limit prescribed in Table 1 shall not exceed if the length and intensity of the stain obtained with the material is not greater than that produced in the control test.

ANNEX G

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF HEAVY METALS (as Pb)

G-1 OUTLINE OF THE METHOD

Heavy metals are determined by comparing the colour produced by the material with hydrogen sulphide solution against that produced by a standard lead solution.

G-2 APPARATUS

G-2.1 Nessler Tubes

50 ml capacity.

G-3 REAGENTS

G-3.1 Concentrated Hydrochloric Acid

See IS 265.

G-3.2 Standard Lead Solution

Dissolve 1.60 g of lead nitrate in water and make up the volume to 1 000 ml. Transfer exactly 10 ml of the solution into a volumetric flask and dilute it again with water to 1 000 ml mark. One millilitre of this solution contains 0.01 mg of lead (as Pb). The dilute solution shall be prepared fresh.

G-3.3 Acetic Acid

Approximately 1 N.

G-3.4 Hydrogen Sulphide Solution

Saturated.

G-4 PROCEDURE

Weigh exactly 1.0 g of the prepared sample and add 50 ml of water. Add 0.5 ml of acetic acid and 10 ml of hydrogen sulphide solution. In the second Nessler tube, carry out a control test using 5 ml of standard lead solution in case of technical grade of the material and one millilitre in case of analytical reagent grade and the same quantities of other reagents. Dilute the contents of each tube to 50 ml and shake well. Compare the colour produced in the two tubes.

G-4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

ANNEX H

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF CHLORIDES (as Cl)

H-1 OUTLINE OF THE METHOD

Chlorides are determined by comparing the opalescence produced by the material with silver nitrate solution, against a dilute chloride solution of known strength.

H-2 APPARATUS

H-2.1 Nessler Tubes

50 ml capacity.

H-3 REAGENTS

H-3.1 Dilute Nitric Acid

5 N approximately.

H-3.2 Silver Nitrate Solution

5 percent (*m/v*).

H-3.3 Standard Sodium Chloride Solution

Dissolve 0.16 g of sodium chloride in 1 litre of water. One millilitre of this solution is equivalent to 0.1 mg of chloride (as Cl).

H-4 PROCEDURE

Dissolve 5 g of the *prepared sample* in water in a Nessler tube and make up the volume to 50 ml. Add one millilitre of dilute nitric acid and one millilitre of silver nitrate solution. Carry out a control test in the other Nessler tube using 1 ml of standard sodium chloride solution and the same quantities of other reagents in the same total volume of the reaction mixture. Stir with glass rod and after 5 min compare the opalescence produced in the tubes.

H-4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced with the material is not greater than that produced in the control test.

ANNEX J

(*Clause* 6.1)

SAMPLING OF POTASH ALUM

J-1 GENERAL REQUIREMENTS OF SAMPLING

J-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

J-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

J-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

J-1.4 The sample shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

J-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

J-2 SCALE OF SAMPLING

J-2.1 Lot

All the containers in a single consignment of the material of one grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

J-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

J-2.2 The number (*n*) of containers to be chosen from a lot shall depend on the size of the lot (*N*) and shall be in accordance with col 1 and 2 of Table 2.

J-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used (*see* IS 4905). In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them 1, 2, 3 ...... up to *r* and so on in one order, where *r* is the integral part of *N*/*n*. Every *r*th container thus counted shall be taken out for drawing samples.

Table 2 Number of Containers to be Selected

(*Clause* J-2.2)

Lot Size (*N*)	Number of Containers to be Selected (*n*)
(1)	(2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and over	7

J-3 TEST SAMPLES AND REFEREE SAMPLE

J-3.1 Preparation of Test Samples

J-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **3** and shall not exceed 1 kg.

J-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

J-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **J-1.5**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

J-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see* **J-3.1.2**) and a set of individual samples (*see* **J-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

J-4 NUMBER OF TESTS

J-4.1 Tests for the determination of potash alum shall be conducted each of the individual samples for all the grades.

J-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

J-5 CRITERIA FOR CONFORMITY

J-5.1 For Individual Samples

The test results for potash alum shall be recorded, and the mean and the range for these test results shall be calculated as follows:

Mean $\bar{x}$ = Sum of the test results divided by the number of test results.

Range (R) = The difference between the maximum and the minimum values of the test results.

The value of expression ($\bar{x}$ - 0.6R) shall be calculated. If the value of this expression is greater than or equal to the minimum specified value for the relevant grade in Table 1, the lot shall be declared to have satisfied the requirements for this characteristic.

J-5.2 For Composite Sample

The test results on the composite sample shall meet the corresponding requirements for the relevant grade.

J-5.3 A lot shall be declared as conforming to this specification if it is satisfies the requirements of the specification for each of the characteristics for the relevant grade.

ANNEX K

(Foreword)

COMMITTEE COMPOSITION

General Inorganic Chemicals Sectional Committee, CHD 03

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Shri Lajinder Singh, Director (Chem)	Director General, BIS (*Ex-officio Member*)

Member Secretary

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Deputy Director (Chem), BIS

Alums and Phosphates Subcommittee, CHD 3:1

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